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During management of the Hanford Single-Shell Waste Tanks (SST), the site speratorio of the supernate as nickel cesium ferrocyanide to allow disposal of the supernate as low-level waste. This freed valuable tank of the supernate as low-level waste generate with nitrate and the supernate as arisen that the supernate as arisen that the supernate as arisen that the supernate arisen with nitrate, another waste component, and/or its radiolysis product nitrite.

Whether such a reaction could occur in the SST's is complicated by the fact that the physical configuration of the ferrocyanide in each of the SST's is unknown. The ferrocyanide could be mixed and/or diluted with other waste solids, which could include bismuth phosphate, sodium aluminate, and hydrous iron oxide. The ferrocyanide could also be present as a layer or pocket mixed intimately with sodium nitrate and/or nitrite.

The current Hanford Principal Contractor, Westinghouse Hanford Company (WHC), has requested that the Pacific Northwest Laboratory (PNL) evaluate the potential for explosive ferrocyanide reactions on a worst case basis. The worst case is believed, at this time, to be a mixture of nickel cesium ferrocyanide and a mixture of nitrate and nitrite without any dilution by inert waste constituents. PNL will perform energetic and small-scale explosion testing, however, additional capabilities are required for large-scale explosion tests. The large-scale explosion test(s) will be performed by Los Alamos National Laboratory (LANL)

#### **Objective**

The objective of the Ferrocyanide Safety Evaluation Program is to determine whether the ferrocyanide could be an explosive hazard under current SST management guidelines. Under these guidelines the SST temperature is maintained at less than 100°C and the solids or sludge is kept moist.

The objective of the Large-Scale Explosion task within the Ferrocyanide Safety Evaluation Program, is to measure the explosive hazard of a mixture of ferrocyanide and a pure nitrate/nitrite mixture with no other waste components present. In this task a series of preliminary small-scale or laboratory tests will be performed to determine handling hazards and to determine the effects of potential initiating events such as friction, spark, and impact sensitivities. The preliminary small-scale tests will be followed by a large-scale test if LANL determines that the test mixture can be handled safely.

#### Scope

The objective of the Large-Scale Explosion task will be met by performing a series of small-scale experiments to determine that the explosion mixture can be handled safely followed by one or more large-scale explosion tests, which will be defined after the small-scale experiments are complete. The small-scale sub-task will include tests to determine (1) impact sensitivity, (2) friction sensitivity, (3) spark sensitivity, (4) thermal properties, (5) gas evolution as a function of temperature, (6) vacuum stability, (7) Henkin Critical Temperature, and (8) adiabatic temperature rise as a function of time.

## Task Definition

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This section describes the activities which will be accomplished in the Large-Scale Explosion task by subtask.

## 1. Preliminary Safe-Handling Experiments

The Preliminary Safe-Handling Experiments will consist of a battery of experiments using a PNL supplied mixture of ferrocyanide and a nitrate and nitrite mix, hereafter identified as the FeCN Mix. LANL will provide the DOT-approved shipping container rated for 25 g of the potentially explosive mixture. The container provided will reduce the shipping classification of experimental explosives and unknown materials from Class A explosives to Class C explosives.

LANL will determine the Impact Sensitivity of the FeCN Mix at ambient conditions. LANL will test the mixture for impact sensitivity by dropping a weight of known mass from a known height onto a sample on sand paper (12A test) and bare metal (12B test). The tester will determine by sight and sound whether the material explodes. This test will provide a comparative ranking of the ease of explosive ignition of FeCN Mix to known explosives such as a sensitive secondary explosive like PETN and insensitive secondary explosives like triamino trinitro-benzene. An explosive with known behavior will be tested prior to FeCN Mix testing to demonstrate that the equipment is functioning properly.

LANL will determine the Friction Sensitivity of the FeCN Mix at ambient conditions. LANL will test the mixture for friction sensitivity by dragging a weighted ceramic pin through a sample of the salt confined on a ceramic plate. The tester will determine by sight and sound whether the material explodes. This test will evaluate the ease of ignition of the FeCN Mix if confined in screw threads or other pinching environments. An explosive with known behavior will be tested prior to testing the FeCN Mix to demonstrate that the equipment is functioning properly.

LANL will determine the Spark Sensitivity of the FeCN Mix at ambient conditions. In this test an electric spark of known energy is forced to traverse the specimen and the amount of reaction is assessed. The purpose of this test is to ensure that static electricity from human body capacitance will not ignite the mixture. An explosive with known behavior will be tested prior to testing the FeCN Mix to demonstrate that the equipment is functioning properly.

LANL will determine Preliminary Thermal Properties. In this test Differential Thermal Analysis (DTA), which measures heat evolution as a function of steadily increasing temperature, will be used to determine the onset temperature of the decomposition exotherm, the rate of exothermic build up, and the nature of the thermal explosion when the sample is lightly confined. A material with known behavior will be tested prior to testing the FeCN Mix to demonstrate that the equipment is functioning properly with respect to temperature calibration and thermal response.

LANL will determine gas evolution as a function of temperature. A thermoconductivity detector will be used to measure total gases evolved which have a different thermoconductivity than helium, such as water, carbon dioxide, and nitrogen, as the sample is heated up at a controlled rate. This test will provide information on the temperature of decomposition of the FeCN Mix. A material with known behavior will be tested prior to testing the FeCN Mix to demonstrate that the equipment is functioning properly.

LANL will measure the Vacuum Stability of the FeCN Mix. This test measures gas evolution as a function of time at 120°C or higher. The sample will be placed in an oil bath, the system evacuated, the sample heated to temperature, and the volume of gas evolved will be measured a sa function of time using a mercury manometer during a 48 hour period. This test is used to evaluate the thermal sensitivity of a material to pressing and handling. The system's volume will be measured or documentation of a recent calibration will be provided.

LANL will perform a Henkin Critical Temperature test of the FeCN Mix. In this test confined samples of known geometry are placed in a molten-metal bath at several different temperatures and the times to explosion measured. The data will be plotted and analyzed to determine the critical temperature. The critical temperature is the temperature where the center of the sample is generating heat faster than it can be dissipated by thermal conduction to the walls of the cell and bath. If differences exist between the DTA exotherm temperature and the critical temperature, effects are indicated that would be of concern in large-scale tests. An explosive with known behavior will be tested prior to testing the FeCN Mix to demonstrate that the equipment is functioning properly.

LANL will perform an Accelerating Rate Calorimetry (ARC) test of the FeCN Mix. This test measure adiabatic temperature rise as a function of time. The sample is confined in a container of known geometry and heated in 5-10°C steps until evidence of exothermic reaction can be detected using a thermocouple. At this point the external heating is stopped and the temperature of the sample is monitored as a function of time. This test will provide the initial reaction temperature of the FeCN Mix and change in rate of heat evolution with temperature. To demonstrate that the equipment is operating properly, an explosive or material with known behavior will be tested prior to testing of the FeCN Mix or documentation of a recent, within 1 year, equipment performance evaluation will be provided.

## 2. Large-Scale Test

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If the Preliminary Safe-Handling Tests, indicate that a large-scale test can be performed safely, a large-scale test(s) will be defined, a cost estimate and schedule provided, and performed pending approval by PNL and WHC and funding by WHC.

## OA Requirements and Change Control

LANL will allow Q-Cleared PNL technical representatives to observe, review, and discuss the work activities and documentation identified in the Statement of Work.

LANL will provide the following documentation:

- 1. Who is responsible for accomplishing the work. As a minimum LANL will provide resume(s) of key technical personnel and an organizational chart for the responsible organization(s). If a subcontractor to LANL performs any work, the same minimum personnel and organizational information will be provided.
- 2. LANL will provide a description of the test method. As a minimum LANL will describe how the test was done, what parameters were measured, and the equipment used to measure the critical parameters. These can be documented in the report. In addition, LANL will provide copies of the procedures used for the tests.
- 3. LANL will provide records of equipment or test calibrations, controls, or verifications used to insure that the test and test equipment is operating properly. Examples are equipment calibration records, documentation of standard laboratory tests used to check equipment operation, and documentation of user calibrations.
- 4. LANL will provide copies of all experimental records. Examples are signed and dated logbook pages detailing activities and data collected. Another option is experimental data sheets that are signed and dated. The operator and cognizant scientist will sign and date all experimental records.
- 5. LANL will document deviations from procedures or problems and their resolution; e.g. correcting weight by known balance error.
- 6. LANL will notify PNL in writing of any needed changes in the scope of work identified in this Statement of Work, any delays in the LANL provided schedule, and any increase in cost. LANL will obtain PNL approval of any changes in scope or increases in cost prior to accomplishing the scope change or cost overrun.
- 7. LANL will document traceability of materials to show that materials used meet procedural requirements; e.g. traceability to NBS if required by procedure, or to manufacturer and lot number for chemical reagents or supply house chemicals.
- 8. LANL will insure traceability of the analytical results to the FeCN Mix received from PNL by assigning the FeCN Mix with a unique identifier and referencing this identifier on all analytical test records and results.

#### Products/Deliverables

The product of the Preliminary Safe-Handling Tests will be a report detailing the results of the tests and an assessment of the test results. The information in the report will be suitable for release to the public without additional approvals. This report will be suitable for inclusion in a formal PNL document.

The product of the Large-Scale Test will be a report detailing the results of the test(s) and an assessment of the test results. The information in the report will be suitable for release to the public without additional approvals. This report will be suitable for inclusion in a formal PNL document.

In addition to the two reports, LANL will provide copies of all the documentation identified in Section 5.0 QA Requirements and Change Control.

All products/deliverables are to be delivered to the PNL Technical Administrator, R. D. Scheele.

#### Responsible Personnel

It is expected the H. H. Cady will act as the task manager and he and other members of the LANL M-1 Explosives Testing Group will serve as Principal Investigators and testers.

#### Schedule and Milestones

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The following schedule and milestones assumes that the Preliminary Safe Handling Experiments will require 4 months from the receipt of funding and sample. This schedule is contingent on LANL, PNL, and WHC reaching agreement on the Draft Statement of Work by December 8, 1989.

Milestone	Estimated Completion Date
LANL, PNL and WHC Technical Personnel reach agreement on draft Statement of Work	December 8, 1989
LANL receives Integrated Contractor Purchase Order from PNL	January 8, 1990
LANL accepts Integrated contractor Purchase Order	January 15, 1990
PNL authorizes funding for LANL to begin Preliminary Handling Experiments	January 17, 1990
LANL receives Reaction Mixture from PNL	January 31, 1990
LANL begins Preliminary Safe Handling Experiments	February 5, 1990 <sup>1</sup>
LANL provides Final Report on Preliminary Safe Handling Experiments to PNL	May 30, 19901
LANL provides Cost and Schedule estimates for Large-Scale Explosion Test(s) to PNL	May 30, 1990 <sup>1</sup>
PNL, WHC, and LANL reach agreement on Large-Scale Experiment	June 29, 1990 <sup>2</sup>
WHC provides funding to PNL for Large-Scale Test(s)	July 18, 1990 <sup>2</sup>
PNL authorizes funds for LANL to begin Large-Scale Test(s)	July 25, 1990 <sup>2</sup>
LANL begins preparing for Large-Scale Explosion Test(	s) August 1, 1990 <sup>2</sup>
LANL receives reactants for Large-Scale Explosion Test(s) from PNL	September 3, 1990 <sup>2</sup>
Final Report on Large-Scale Explosion Test(s) to PNL	October 15, 1990 <sup>2</sup>

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<sup>1.</sup> Date contingent on receipt of sample and approval to spend funds at LANL.

<sup>2.</sup> Tentative date to be revised after LANL completes preliminary testing.

# Budget and Spend Plan

The estimated total LANL budget for the Preliminary Small-Scale Safety Study is \$69K. The estimated spend plan is:

January 1990		\$ 5K
February 1990		\$14K
March 1990		\$14K
April 1990		\$17K
May 1990		\$17K
June 1990		\$ 2K
	TOTAL	\$69K

## Deliverables

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One copy of all deliverables shall be submitted to the Battelle Technical Administrator, Randy Scheele.